

# FACTORS RELATED TO THE FLAVOR STABILITY DURING STORAGE OF FOAM-DRIED WHOLE MILK. III. EFFECT OF ANTIOXIDANTS

A. TAMSMA, T. J. MUCHA, AND M. J. PALLANSCH

Dairy Products Laboratory, Eastern Utilization Research and Development Division, USDA  
Washington, D. C.

## SUMMARY

The storage stability of vacuum foam-dried whole milk powder containing antioxidants was studied organoleptically. The relative effectiveness of antioxidants in reducing flavor deterioration in stored samples was found to decrease in the following order: lauryl gallate, propyl gallate, nordihydroguaiaretic acid, ascorbyl palmitate, butylated hydroxy anisole, ascorbic acid, dihydroquercetin, sodium diethyldithiocarbamate, thiodipropionic acid, quercetin, dilaurylthiodipropionate.

Largest flavor stabilizing effects in samples stored at 80 F for six months were observed in air packs. Statistical analysis of the data showed that only dihydroquercetin, ascorbic acid, ascorbyl palmitate, or sodium diethyldithiocarbamate produced significant improvement of the flavor scores of powders packed in nitrogen containing 0.1 or 1.0% oxygen.

None of the antioxidants tested allowed the production of powders with greater stability than those obtained by drying milk heated at 165 F for 30 min.

Stabilization of the initial flavor of whole milk powders produced by drying foamed concentrates under high vacuum (7) is one of the objectives of research carried out in our Laboratory. Previous papers from this group have demonstrated that the flavor stability of this material could be increased by packaging the finished product in inert gas (10) or heat-treating milk before drying (9). Continued investigation of the flavor stability problem has resulted from objections to the low levels of oxygen required for adequate packaging, and cooked flavor in powders made from milk heated in excess of pasteurization requirements.

This paper reports our observations of the effects of added antioxidants on the flavor stability of foam-dried whole milk. To check for the possible existence of stabilizing combinations, a few antioxidants were added to heat-treated milk before drying, and samples of all experimental powders were packed in sealed tins containing graded levels of oxygen.

The valuation of the type and intensity of flavors in stored samples was limited to organoleptic methods by the lack of suitable chemical tests (6).

Precedent for this work has been established by other authors' studies of the flavor-stabiliz-

ing effect of antioxidants in spray-dried whole milk powders (1, 3, 4, 8, 11).

## MATERIALS AND METHODS

Possible variation in the composition of the milk supply during the course of the work was reduced by using mixed-herd milk from cows maintained on an invariant husbandry regime at the Agricultural Research Center at Beltsville, Maryland.

The antioxidants used in this study, lauryl gallate (LG), propyl gallate (PG), nordihydroguaiaretic acid (NDGA), ascorbyl palmitate (AP), butylated hydroxy anisole (BHA), ascorbic acid (AA), dihydroquercetin (DQ), sodium diethyldithiocarbamate (SDDC), thiodipropionic acid (TDPA), quercetin (Q), and dilaurylthiodipropionate (DLTDP) were all obtained from commercial sources. The study was not restricted to additives approved for food use by the Food and Drug Administration.

Experimental samples of whole milk powder were prepared from standardized milk, pasteurized by holding at 145 F for 30 min, except where noted. The milk was concentrated to 50% solids, homogenized, injected with nitrogen, and dried in the form of a foam under high vacuum.

Powder was produced by breaking the dried foam through a 20-mesh screen. Samples of

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each powder were sealed in tins containing: (a) air; (b) nitrogen plus 1.0% O<sub>2</sub>; (c) nitrogen plus 0.1% O<sub>2</sub>. The packaged powders, containing 2.5% moisture and 26% fat, were stored at 80 F.

Antioxidants, added at a level to give a concentration of 0.01% in the dry product (except ascorbic acid 0.3%, ascorbylpalmitate 0.5%), were dissolved in a suitable solvent and stirred into the concentrate just before homogenization. Alcohol, propylene glycol, and water were used as solvents in volumes equal to 0.25% of the concentrate to which they were added. Similar volumes of the same solvent were added to the controls in each series.

The type and intensity of the flavors in the reconstituted samples studied were determined by a panel of ten trained judges. Sensory experience was converted into flavor scores by use of a standard score-card. The significance of the scores was evaluated by use of statistical methods based on the analysis of variance.

Specific details concerning all the above-mentioned operations have been previously reported (9).

#### RESULTS

In consideration of the facts that a variety of antioxidant solvents were used in the study, and that the preparation of experimental samples extended over a considerable time interval,

each group of samples made from a single lot of milk was analyzed as a separate entity. Where significance was found, using the analysis of variance, a mean comparison was made with Duncan's Multiple Range test. In the tables showing antioxidant/storage time interaction the mean flavor scores (MFS) are obtained by averaging data from all air and gas packs. Likewise, the MFS in the antioxidant/oxygen level table are obtained by averaging the flavor scores of the two-, four-, and six-months-old samples.

Tables 1 through 3 present the mean separation or groupings resulting from the employment of Duncan's Multiple Range test. In each related group in the tables those mean scores having a common letter in the ranking column are statistically indistinguishable.

Data pertaining to the storage behavior of vacuum foam-dried powders made from milk with minimum pasteurization are presented in Tables 1 and 2. Evidence for mean flavor score improvement by the inclusion of antioxidants is most easily observed in air pack data. Inclusion of LG, AP, and NDGA effected the greatest flavor stabilization of powders packed in air. The mean flavor score of all six-months-old packs of powder containing LG was statistically indistinguishable from the mean flavor score of all two-months-old controls in the series.

TABLE 1

Rank comparison of mean flavor scores of whole milk powders containing various antioxidants (milk pasteurized 145 F 30 min)

| Interaction of<br>antioxidant and storage time |                       |                   |                 | Interaction of<br>antioxidant and O <sub>2</sub> level in pack |                                  |                   |                 |
|--|-----------------------|-------------------|-----------------|--|----------------------------------|-------------------|-----------------|
| Anti-oxidant                                   | Storage time (months) | Mean flavor score | Rank (5% level) | Anti-oxidant   | In-pack O <sub>2</sub> level (%) | Mean flavor score | Rank (5% level) |
| PG   | 2                     | 37.04             | A               | LG   | 0.1                              | 36.81             | A               |
| LG   | 2                     | 36.75             | A               | Control  | 0.1                              | 36.69             | AB              |
| LG   | 6                     | 35.75             | B               | PG   | 0.1                              | 36.57             | AB              |
| LG   | 4                     | 35.58             | B               | LG   | 1.0                              | 36.36             | ABC             |
| Control  | 2                     | 35.19             | BC              | PG   | 1.0                              | 36.17             | BC              |
| PG   | 6                     | 34.95             | CD              | Control  | 1.0                              | 35.86             | C               |
| PG   | 4                     | 34.52             | D               | LG   | Air                              | 34.91             | D               |
| Control  | 6                     | 34.46             | D               | PG   | Air                              | 33.77             | E               |
| Control  | 4                     | 34.38             | D               | Control  | Air                              | 31.48             | F               |
| Q  | 2                     | 34.83             | A               | DQ   | 1.0                              | 35.48             | A               |
| DQ   | 2                     | 34.59             | B               | DQ   | 0.1                              | 35.45             | A               |
| DQ   | 4                     | 34.56             | B               | Control  | 0.1                              | 35.20             | B               |
| Control  | 2                     | 34.24             | C               | Q  | 0.1                              | 35.05             | B               |
| Control  | 4                     | 34.08             | C               | Q  | 1.0                              | 34.74             | C               |
| Q  | 4                     | 33.93             | CD              | Control  | 1.0                              | 34.59             | C               |
| DQ   | 6                     | 33.74             | DE              | Q  | Air                              | 32.30             | D               |
| Control  | 6                     | 33.54             | EF              | Control  | Air                              | 32.07             | E               |
| Q  | 6                     | 33.33             | F               | DQ   | Air                              | 31.96             | E               |

TABLE 2

Rank comparison of mean flavor scores of whole milk powders containing various antioxidants  
(milk pasteurized 145 F 30 min)

| Interaction of<br>antioxidant and storage time |                       |                   |                 | Interaction of<br>antioxidant and O <sub>2</sub> level in pack |                                  |                   |                 |
|--|-----------------------|-------------------|-----------------|--|----------------------------------|-------------------|-----------------|
| Anti-oxidant                                   | Storage time (months) | Mean flavor score | Rank (5% level) | Anti-oxidant   | In-pack O <sub>2</sub> level (%) | Mean flavor score | Rank (5% level) |
| BHA  | 2                     | 35.39             | A               | Control  | 0.1                              | 35.42             | A               |
| BHA  | 4                     | 35.10             | A               | NDGA   | 0.1                              | 35.11             | A               |
| Control  | 2                     | 34.94             | A               | NDGA   | 1.0                              | 35.10             | A               |
| Control  | 4                     | 34.74             | A               | Control  | 1.0                              | 35.02             | A               |
| NDGA   | 2                     | 33.72             | B               | BHA  | 0.1                              | 34.75             | A               |
| Control  | 6                     | 33.48             | B               | NDGA   | Air                              | 33.76             | B               |
| BHA  | 6                     | 33.48             | B               | BHA  | 1.0                              | 33.07             | BC              |
| NDGA   | 4                     | 33.28             | B               | Control  | Air                              | 32.71             | C               |
| NDGA   | 6                     | 32.70             | B               | BHA  | Air                              | 31.88             | D               |
| Control  | 2                     | 34.77             | A               | Control  | 0.1                              | 34.90             | A               |
| TDPA   | 2                     | 34.15             | AB              | DLTDP  | 0.1                              | 34.83             | A               |
| DLTDP  | 2                     | 33.97             | B               | Control  | 1.0                              | 34.78             | A               |
| Control  | 4                     | 33.95             | B               | TDPA   | 1.0                              | 34.31             | A               |
| TDPA   | 4                     | 33.65             | BC              | TDPA   | 0.1                              | 34.16             | A               |
| DLTDP  | 4                     | 33.50             | BC              | DLTDP  | 1.0                              | 33.21             | B               |
| Control  | 6                     | 32.83             | CD              | Control  | Air                              | 31.87             | C               |
| TDPA   | 6                     | 32.48             | D               | TDPA   | Air                              | 31.66             | C               |
| DLTDP  | 6                     | 31.98             | D               | DLTDP  | Air                              | 31.55             | C               |
| AA   | 2                     | 35.13             | A               | SDDC   | 0.1                              | 35.54             | A               |
| AP   | 2                     | 35.04             | A               | SDDC   | 1.0                              | 35.47             | AB              |
| Control  | 2                     | 34.76             | AB              | AA   | 0.1                              | 35.14             | ABC             |
| AP   | 6                     | 34.44             | BC              | AP   | 0.1                              | 34.93             | BCD             |
| SDDC   | 2                     | 34.13             | CDE             | AP   | 1.0                              | 34.76             | CD              |
| AP   | 4                     | 33.94             | CDEF            | AA   | 1.0                              | 34.50             | CD              |
| AA   | 4                     | 33.84             | DEF             | Control  | 0.1                              | 33.85             | E               |
| Control  | 4                     | 33.77             | EF              | AP   | Air                              | 33.73             | E               |
| SDDC   | 4                     | 33.52             | F               | Control  | 1.0                              | 33.55             | E               |
| SDDC   | 6                     | 33.38             | FG              | AA   | Air                              | 32.21             | F               |
| AA   | 6                     | 32.88             | G               | Control  | Air                              | 31.14             | G               |
| Control  | 6                     | 30.00             | H               | SDDC   | Air                              | 30.01             | H               |

TABLE 3

Rank comparison of mean flavor scores of whole milk powders containing various antioxidants  
(milk heated 165 F 30 min)

| Interaction of<br>antioxidant and storage time |                       |                   |                 | Interaction of<br>antioxidant and O <sub>2</sub> level in pack |                                  |                   |                 |
|--|-----------------------|-------------------|-----------------|--|----------------------------------|-------------------|-----------------|
| Anti-oxidant                                   | Storage time (months) | Mean flavor score | Rank (5% level) | Anti-oxidant   | In-pack O <sub>2</sub> level (%) | Mean flavor score | Rank (5% level) |
| Control  | 4                     | 36.84             | A               | BHA  | 0.1                              | 36.60             | A               |
| NDGA   | 4                     | 36.84             | A               | BHA  | 1.0                              | 36.36             | A               |
| BHA  | 4                     | 36.71             | AB              | Control  | 0.1                              | 36.33             | A               |
| NDGA   | 2                     | 36.25             | ABC             | NDGA   | Air                              | 36.26             | A               |
| BHA  | 2                     | 36.06             | ABC             | Control  | 1.0                              | 36.06             | A               |
| Control  | 2                     | 36.03             | ABC             | Control  | Air                              | 36.04             | A               |
| NDGA   | 6                     | 35.67             | BC              | NDGA   | 0.1                              | 36.02             | A               |
| Control  | 6                     | 35.67             | BC              | NDGA   | 1.0                              | 35.85             | A               |
| BHA  | 6                     | 35.36             | C               | BHA  | Air                              | 35.80             | A               |

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Even though SDDC significantly lowered the mean flavor score of air-packed samples, its inclusion actually raised the mean flavor score of gas-packed samples well above those of the matching controls. Elevation of mean flavor scores of samples in gas-packed samples above those of the appropriate control was also observed with powders containing AA, AP, and DQ. All other antioxidants were ineffective in gas packs.

Data pertaining to the flavor-stabilizing effect of antioxidants added to heat-treated milk before drying are found in Table 3. Even though the analysis of variance indicated interaction between storage time, oxygen levels, and antioxidants, little significant difference could be detected between the mean flavor scores of all samples.

The mean flavor score of all six-months-old controls made from milk heated at 165 F for 30 min was almost identical to that of the best six-months-old antioxidant-containing series made from milk receiving minimum heat treatment.

Mean flavor scores of powder made from milk that had received high heat treatment were not raised by inclusion either of NDGA or of BHA. Heat treatment was capable of reducing the difference between air and gas packs to insignificant levels.

Whereas an analysis of mean flavor scores is relatively informative about the effect of antioxidants on the loss of fresh flavor in whole milk powder during storage, an examina-

tion of the response of the judges in terms of the flavors detected is necessary to define the actual flavor changes encountered during storage.

Raw data pertaining to the individual panel members' judgments of the type of flavors which determined the flavor scores of the samples have been consolidated in Tables 4 and 5. These tables show the effect of storage time and in-pack O<sub>2</sub> level on the flavors detected in all packs of each type of powder. The flavors listed on the score-card have been grouped, in a somewhat arbitrary fashion, to save space. Results are reported in terms of the per cent of total judgments falling in each flavor group.

Recourse to the raw data shows that the increased percentages of astringent plus foreign flavors found in powders containing gallates, NDGA, or AA resulted primarily from increased detection of astringency. Very few judges detected foreign flavors in any of the samples studied. The maximum number, 6% of the total, was associated with the powder containing AP.

The varying ability of the added antioxidants to preserve the sulphydryl groups released during pasteurization may account for the observed differences in the cooked + feed group, since all results tabulated were obtained from a study of powders made from milk pasteurized at minimum standards. The feed flavors reported never exceeded 5% of the total judgments pertaining to the samples and varied little among the powders containing different antioxidants.

TABLE 4  
Effect of storage time on type of flavor found in experimental whole milk powders  
(reported as per cent total judgment summing 0.1% O<sub>2</sub>, 1.0%, and air packs)

| Anti-oxidant | Principal type of flavor detected |    |    |                       |    |    |                       |    |    |                       |    |    |
|--------------|-----------------------------------|----|----|-----------------------|----|----|-----------------------|----|----|-----------------------|----|----|
|              | Astringent + foreign              |    |    | Cooked + feed         |    |    | Oxidized + stale      |    |    | All others            |    |    |
|              | Storage time (months)             |    |    | Storage time (months) |    |    | Storage time (months) |    |    | Storage time (months) |    |    |
|              | 2                                 | 4  | 6  | 2                     | 4  | 6  | 2                     | 4  | 6  | 2                     | 4  | 6  |
| Control      | 13                                | 11 | 6  | 9                     | 7  | 6  | 64                    | 71 | 82 | 14                    | 11 | 6  |
| NDGA         | 20                                | 24 | 0  | 24                    | 10 | 10 | 36                    | 46 | 76 | 20                    | 20 | 14 |
| BHA          | 7                                 | 17 | 0  | 16                    | 6  | 13 | 40                    | 47 | 81 | 37                    | 30 | 6  |
| LG           | 30                                | 17 | 17 | 13                    | 10 | 17 | 47                    | 50 | 49 | 10                    | 23 | 17 |
| PG           | 23                                | 17 | 7  | 10                    | 7  | 24 | 40                    | 56 | 56 | 27                    | 20 | 13 |
| Q            | 10                                | 7  | 10 | 7                     | 0  | 0  | 67                    | 76 | 83 | 16                    | 17 | 7  |
| DQ           | 20                                | 10 | 10 | 3                     | 10 | 3  | 47                    | 73 | 84 | 30                    | 7  | 3  |
| TDPA         | 13                                | 6  | 3  | 10                    | 3  | 3  | 63                    | 84 | 94 | 14                    | 7  | 0  |
| DLTDP        | 7                                 | 13 | 0  | 7                     | 0  | 10 | 73                    | 87 | 90 | 13                    | 0  | 0  |
| AA           | 13                                | 10 | 3  | 16                    | 17 | 6  | 57                    | 67 | 77 | 14                    | 6  | 14 |
| AP           | 20                                | 27 | 20 | 17                    | 7  | 10 | 43                    | 63 | 50 | 20                    | 3  | 20 |
| SDDC         | 3                                 | 3  | 3  | 14                    | 17 | 3  | 77                    | 77 | 84 | 6                     | 3  | 10 |

TABLE 5

Effect of in-pack O<sub>2</sub> level on type of flavor found in experimental whole milk powders (reported as per cent total judgments summing two, four, and six months storage periods)

| Anti-oxidant | Principal type of flavor detected |     |     |                              |     |     |                              |     |     |                              |     |     |
|--------------|-----------------------------------|-----|-----|------------------------------|-----|-----|------------------------------|-----|-----|------------------------------|-----|-----|
|              | Astringent + foreign              |     |     | Cooked + feed                |     |     | Oxidized + stale             |     |     | All others                   |     |     |
|              | In-pack O <sub>2</sub> level      |     |     | In-pack O <sub>2</sub> level |     |     | In-pack O <sub>2</sub> level |     |     | In-pack O <sub>2</sub> level |     |     |
|              | 0.1                               | 1.0 | Air | 0.1                          | 1.0 | Air | 0.1                          | 1.0 | Air | 0.1                          | 1.0 | Air |
| Control      | 12                                | 12  | 6   | 12                           | 7   | 3   | 60                           | 70  | 85  | 16                           | 11  | 6   |
| NDGA         | 10                                | 23  | 9   | 20                           | 10  | 13  | 50                           | 53  | 57  | 20                           | 14  | 21  |
| BHA          | 13                                | 3   | 6   | 17                           | 13  | 7   | 50                           | 61  | 57  | 20                           | 23  | 30  |
| LG           | 27                                | 23  | 13  | 20                           | 10  | 10  | 33                           | 47  | 67  | 20                           | 20  | 10  |
| PG           | 10                                | 23  | 14  | 17                           | 10  | 13  | 43                           | 47  | 63  | 30                           | 20  | 10  |
| Q            | 17                                | 10  | 0   | 7                            | 0   | 0   | 70                           | 66  | 90  | 6                            | 24  | 10  |
| DQ           | 10                                | 17  | 13  | 6                            | 6   | 3   | 64                           | 64  | 74  | 20                           | 13  | 10  |
| TDPA         | 7                                 | 10  | 7   | 10                           | 6   | 0   | 73                           | 74  | 93  | 10                           | 10  | 0   |
| DLTDP        | 10                                | 3   | 7   | 13                           | 3   | 0   | 77                           | 84  | 90  | 0                            | 10  | 3   |
| AA           | 10                                | 10  | 7   | 13                           | 17  | 10  | 63                           | 60  | 77  | 14                           | 13  | 6   |
| AP           | 30                                | 17  | 20  | 14                           | 14  | 7   | 50                           | 53  | 54  | 6                            | 16  | 19  |
| SDDC         | 3                                 | 7   | 3   | 17                           | 10  | 0   | 74                           | 64  | 94  | 6                            | 13  | 3   |

The raw data used to calculate the figures presented in the oxidized + stale group demonstrated the extreme difficulty in obtaining good agreement among taste panel members when samples containing both oxidized and stale flavors were encountered. In general, a rough relationship was observed in which the per cent of stale judgments increased as the oxidized judgments decreased. By combining these two flavor categories, as indicated in the tables, a more consistent flavor pattern was established. All powders were judged to increase in oxidized + stale flavor as storage time increased.

The effectiveness of an antioxidant to maintain fresh flavor in milk should be inversely proportional to the total number of judges finding this oxidized + stale flavor in stored samples. A crude ordering of antioxidant effectiveness can then be established from the reciprocal of the total percentage of judgments falling in this flavor group. Using the data in Table 4 for calculations of this type indicates that the relative ability of added antioxidants to stabilize the flavor of vacuum foam-dried whole milk, averaged over all in pack oxygen levels, decreases as follows: LG > PG > NDGA > AP > BHA > AA > DQ > SDDC > TDPA > Q > DLTDP.

A tabulation of the effect of in-pack oxygen levels on per cent of judgments in the described flavor groups did not reveal flavor change patterns different from those already published concerning the effect of in-pack oxygen levels on flavor scores (10). While the per cent judgments in the stale + oxidized

class decreased with the oxygen content of the pack, it was relatively high in all stored powders regardless of in-package gas composition. Those antioxidants found to increase the flavor stability of gas packs apparently did so by reducing the intensity of oxidized + stale flavor rather than by decreasing the number of judgments in this flavor category.

#### DISCUSSION

This study of the relative ability of various antioxidants to suppress the appearance of oxidized flavor in vacuum foam-dried milk during storage agrees, where comparison is possible, with results obtained by other investigators from studies using spray-dried powders.

Like Abbot and Waite (1), we found lauryl gallate to be the most effective of the antioxidants tested. The astringency induced in milk by inclusion of this material as noted by Tollenaar (11) was picked up by our panel, but the low level of the ten-man response suggested that this flavor could not be considered a major defect that would curtail the use of this antioxidant.

The increased effectiveness of higher homologues of known antioxidants, as demonstrated by the gallates in spray-dried milk (11), was again found in our study. The increased solubility in fat resulting from the added chain length may explain the observed increase in antioxidant power, but a more inviting theory is that the decreased polarity of the long-chain molecules causes them to concentrate at the surface of the fat globules where their presence makes the effective contribution to fat phase

stability. Much additional information is needed on the manner in which antioxidants added to milk distribute themselves and the effect of processing on this distribution.

#### CONCLUSIONS

From the information presented in this paper we have concluded that the mean flavor score of air-packed vacuum foam-dried whole milks stored at 80 F can be considerably improved by inclusion of lauryl gallate during processing. All other antioxidants tested were less effective. The mean flavor scores of gas-packed samples could be slightly improved by use of DQ, AA, AP, or SPDC as an additive.

None of the antioxidants produced samples whose change during six months of storage at 80 F could not be detected by a panel of trained judges, and none of the antioxidants tested proved superior to optimum heat treatment of milk prior to drying.

The consumer acceptance of slight oxidized, slight stale, and definite cooked is undergoing investigation at the present time. Results obtained from this study should aid in determining the practical application of the results presented in this and our previous papers on factors influencing the flavor stability of foam-dried milk.

The close relationship between the physical and chemical properties of powders studied in our papers and those powders now produced in a continuous fashion by either the vacuum belt (2) or atmospheric spray dry process (5) should facilitate translation of our results to the flavor stability problems of those materials.

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